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ARMY RESEARCH OFFICE

RESEARCH AND TECHNOLOGY TRANSFER ION IMPLANTATION TECHNOLOGY FOR SPECIALTY MATERIALS

PROCEEDINGS OF A JOINT WORKSHOP

KNOXVILLE, TN.
OCTOBER 26-27, 1989

ARMY RESEARCH OFFICE
OFFICE OF NAVAL RESEARCH
UNIVERSITY OF TENNESSEE, KNOXVILLE
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13. ABSTRACT (*Maximum 200 words*)

The ion implantation research and technology transfer workshop brought together a diverse group of academic, industrial and government participants. Several key issues highlighted were:

1. A need exists for new technology transfer infrastructures between universities, research labs and industry.
2. Ion implantation technology has promise for several Army and industry applications because of environmental concerns and technological benefits.
3. The U.S. ion implantation industry is primarily service oriented.
4. The cost of Ion implantation technology could be significantly reduced if larger scale production equipment was available for on-line processing. A need exists in the USA for mechanisms and funds to develop such equipment. Such equipment is being implemented by our International competitors (i.e. Panasonic in Japan is currently implanting approximately 3 foot wide low carbon steel razor blade stock continuously).

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Several examples of successful technology transfer mechanisms were presented where initial small amounts of government research funds were significantly leveraged.

RESEARCH AND TECHNICAL TRANSFER OF ION IMPLANTATION TECHNOLOGY FOR SPECIALTY METALS

WORKSHOP PROCEEDINGS

Knoxville, TN
October 26-27, 1989

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EXECUTIVE SUMMARY

The ion implantation research and technology transfer workshop brought together a diverse group of academic, industrial and government participants. Several key issues were highlighted. These include the following:

1. A need exists for new technology transfer infrastructures between universities, research labs and industry.

2. Ion implantation technology has promise for several Army and industry applications because of environmental concerns and technological benefits.

3. The U.S. ion implantation industry is primarily service oriented.

4. The cost of Ion implantation technology could be significantly reduced if larger scale production equipment was available for on-line processing. A need exists in the USA for mechanisms and funds to develop such equipment. Such equipment is being implemented by our International competitors (ie. Panasonic in Japan is currently implanting approximately 3 foot wide low carbon steel razor blade stock continuously).

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Several examples of successful technology transfer mechanisms were presented where initial small amounts of government research funds were significantly leveraged. A detailed summary of the workshop is included with these proceedings.

ROBERT R. REEBER

ACKNOWLEDGEMENTS

The consensus reached at this workshop would not have been possible without the persistence and dedication of Professor Reece Roth who by his thorough coordination of arrangements with the hotel, the University of Tennessee, Knoxville and the invited speakers provided us with an excellent atmosphere to carry out the goals of the meeting. The help of Mrs. Mary Jane Mink, ARO MS Division was invaluable in getting all of the speaker invitation letters out in a timely manner as well as for preparing the honoraria requests. The Machine Tool Builders' Association and National Center for Manufacturing Sciences help in disseminating advance information on the meeting through their newsletter coverage was also appreciated. This significantly increased supplier and user industry participation. Additional financial support from the Office of Naval Research (Dr. George Yoder), Martin Marietta Corporation as Operator of Oak Ridge National Laboratory, and the University of Tennessee, Knoxville (Dr. Thomas Collins) is acknowledged with thanks. Editing and preparing the Workshop summary is always a difficult task. I am especially grateful to the four senior participants who critiqued my summaries of the meeting and minutes of the panel report. Their inputs and corrections helped greatly to make this document a balanced and comprehensive report.

WORKSHOP SUMMARY

After a welcome to the Knoxville area Dr. Thomas C. Collins, Vice Provost for Research at the University of Tennessee, gave a short summary of U.S. problems with technology transfer. The gap that exists between universities and research labs engaged in research and early development and industry, which does not get directly involved until after there is a product to test and evaluate, tends to add a number of years in the technology transfer from university to industry and vice versa. Until we set up a series of infrastructures to fit into this gap, we will always have a competitiveness problem in respect to the rest of the industrial world. It is programs such as these being developed by ARO, and in particular by Bob Reeber, which are helping move infrastructures into this area. Following these introductory remarks Dr. Robert Reeber, Workshop chairman, reviewed meeting objectives and gave highlights of the Army Research Office Program on Degradation, Reactivity and Protection. He briefly discussed his earlier experience with technology transfer involving Department of Energy research programs on geothermal well cements and high temperature elastomeric seals. The key elements for rapid technology transfer (commercial products within 5 to 7 years) were indicated as (1) involvement of appropriate technical industry representatives in early stages of the program, and (2) having some interactions between academic and industrial experts as well as government managers occurring through a formal mechanism such as professional society task group meetings (ASTM, ASME, API, etc.). The lesson learned was that the task groups (because of mutual self interest) were a source of "corporate memory" that continued the standards development through to completion. This provided significant leverages of relatively small amounts of initial government research funding. As a consequence an acceptable benchmark of improved performance was established that could be specified by the government or other purchaser of the technology.

Programmatically the ARO objective for ion implantation research is to lower costs of the technology. Additionally, Dr. Reeber emphasized that the meeting should provide industry (suppliers and users), researchers, and government with a forum for identification of specifics required for further improved technology transfer. In that light, the 60+ participants represented a cross section of the various sectors. The broad participation had been achieved through the cooperation of and newsletter coverage by the National Machine Tool Builders Association (NMTBA) and the National Center for Manufacturing Science (NCMS). Over 160 invitations to the ASME Tool Bit Committee membership as well as appropriate other government and industry supplemented this publicity.

In the first invited paper, Dr. Dearnaley reviewed several technical advantages of ion implantation including:

(1) Corrosion improvements for steel;
(a) Ta implantation of 9310 steel,
(b) N_2 implantation of steel (leading to a 2 orders of magnitude reduction of corrosion for some situations).

(2) Beneficial effects on ceramics;
(a) analogous to shot peening for generation of beneficial surface compressive stresses,
(b) closes surface microcracks thereby reducing initiation sites for mechanical failure.

(3) Control of surface chemistry;
(a) allowing efficient uses of additives that can dispersion harden the surface (borides, nitrides and carbides),
(b) improving wear resistance by strengthening, reducing friction and modifying the run-in,
(c) not being constrained by equilibrium factors such as solubility.

(4) Reduced hydrogen penetration of Ti and steels by ion implanted platinum followed by ion beam mixing with argon.

Work was cited where implanted tools for cam cutters performed two and a half million operations before replacement, or two and a half times longer than unimplanted tools. For this operation individual tool cost was $ 30,000/tool. In other work a more complex ion-beam-mixing treatment (yttrium/nitrogen) increased initial savings from only nitrogen implantation by a factor of 25. It was indicated that costs could be further reduced by increasing ion beam system sizes and by introducing plasma-enhanced ion beam implantation. For some situations ion beam mixing and ion beam coating processes are more appropriate.

Dr. J. Reece Roth, the University of Tennessee at Knoxville, and Dr. John Conrad, University of Wisconsin at Madison, followed up in their talks with a more detailed explanation of the Plasma Source Implantation (PSII) technique. They indicated that PSII has several advantages and disadvantages vis-a-vis line of sight implantation. The advantages include good dose uniformity as well as potential production rates substantially greater than state of the art ion implantation. The disadvantages are that one cannot mass analyze the plasma near the workpiece and non-gaseous ion species are more difficult to generate in PSII. However the group at the University of Wisconsin is actively involved in modifying their PSII device to operate in ion beam mixing modes. Such a modification will allow treatment with metallic ions.

Dr. Ian Brown has a high current ion source under development The work, a spinoff of plasma research, has the potential for implantation of refractory and other heavy metals at reasonable costs. He discussed how costs can be reduced, making exotically alloyed surfaces practical. Applications have already been pursued relating to tribology, corrosion, and the fine tuning of High-Tc superconductivity films. It was interesting to note that practical application of this technology was made possible by Nippon Steel's order and purchase of the first system from a small California company.

Applications for ion beam technology and the results of research were reviewed by Dr. Jim Williams of ORNL. Potential benefits exist for the corrosion inhibition of uranium. He also pointed out that many elements were not beneficial for reducing the corrosion of uranium. Both phosphorus and silicon do seem to be useful in this application. Ion implantation is increasing the life of other parts significantly. It has been useful for smoothing and cleaning of ceramic optics etc. Dr. Williams gave some details about medical implantation. The orthopedic implant business in the US is already at the $5 Billion/yr. level, (250,000 patients at $20,000 per surgery) with the cost of the devices making up about 10% of the bill. It is estimated that another 50,000 implanted devices/year are being done in the UK.

Dr. Graham Hubler continued the research review and summarized previous and current extensive work performed at the Naval Research Laboratory. Their program is 50% research with the remaining 50% of funding coming from external applied program areas. One of the most interesting results reported related to heavy metal implantation for wear resistance of bearing steels. It was found that, although Ti and Ta both lower the coefficient of friction of bearing steels after implantation, Ta was better as it also improved the corrosion resistance. Chromium on the other hand had no effect on the coefficient of friction. The caution was given that one must understand the total materials problem before specifying a treatment. In the course of these talks several important references were cited:

(1) Phys. Stat Sol. A112 16 Mar 89 P. 353-356
(2) Mat. Sci. and Eng. A115 (1989) p. 229-244
(3) Surface Alloying ASM, edited by Hirvonen et al.
(4) PVRL review of MANTECH program NRL Report No. 5928.
(5) British Plastics and Rubber July/Aug. 89.

Mr. Ray Bricault then discussed Spire Corporation's ion implantation production and quality control experience. They have capabilities to carry out mass spectrographic analysis, Rutherford BackScattering Spectrometry (RBS) and other quality control procedures during the implant process. Cheaper parts can

be inexpensively monitored through microhardness measurements of small coupons that are simultaneously ion-implanted during production.

The audience asked many questions during the course of the morning review of research. The perspective of the potential technology user was clear. They were interested in understanding: (1) How to use it?, (2) What is new or different?, (3) What has been done previously?, (4) What are potential problems to watch out for?, (5) What are potential advantages?, (6) What is the best method for checking it out for a specific application?, and (7) Is it worth the effort (ie. are capital costs too high)? The last factor combined with high interest rates was especially important for return on investment strategies. The fact that many ion implantation technology benefits show up primarily from life cycle cost savings means that a significant track record of technical data and product testing is required before any return can be realized. Since the product for such testing also has to be initially produced by more expensive job lot batches, there is a significant financial barrier up front.

During the late afternoon session on the 26th of October the meeting content shifted to a review of Army experience at Corpus Christi Army Depot and Rock Island Arsenal. Dr. Robert Culbertson, Mr. Al Gonzales, and Dr. Lewis Neri reviewed ion implantation tests and helicopter needs as identified at Corpus Christi Army Depot. Tools from the depot sent to MTL for implantation indicated 150 to more than 400% improvements in double blind tests. One important means of technology transfer highlighted by Dr. Culbertson concerned Depot Maintenance Work Requirements (DMWRs). Rather than wait for new standards to be developed and revised through the whole R,D,E and procurement system the DMWR's can be required for remanufacturing. This allows some insertion early on.

It was clear that although ion implantation technology has been promising for several applications it was technologically challenging to transfer. It is not appropriate for every application, and its cost effectiveness will often require the concurrent development of expensive production scale equipment. In the discussion of this paper it was pointed out that Japan was already cost-effectively continuously implanting 3 foot wide strips of low carbon steel for inexpensive razor blades. On the other hand the U.S. ion implantation industry, being comprised of many service-oriented small businesses, does not have the capital up front to develop such specialty production equipment. An analogy was made to expensive lithography equipment developed by Perkin-Elmer and IBM. Perkin Elmer was currently on the auction block with sale of the technology to a Japanese firm highly

likely. The point was made that unless other larger firms got involved much of the production equipment required in 10 years by U.S. industry would be made overseas. This conceivably could be a multibillion dollar market.

The review of the NRL-Spire MANTECH program stimulated the audience to express a range of views. One perspective appeared to be that the program was only partially if at all successful in getting ion implantation technology launched into Navy applications. In contrast the Spire representative felt that much had been accomplished with the groundwork for later efforts established. He felt that the research had definitely aided the development of improved engine bearings for NASA's space shuttles. The discussions involving industrial participants indicated that one of the problems was that it took an exceedingly long time to acquire an adequate data base before management was convinced and would proceed with capital investments for equipment and development. This to some extent set the stage for Dr. Jack Moriarty who described his "freeze frame" method for comprehensively evaluating new tools and cutting procedures. His work at Rock Island has developed a database on tools during machining operations. This has been accomplished by modification of numerically controlled machines so that data from the tools can be continuously retrieved. The various parameters recorded are then related to tool condition and performance. Once a database is established for a few tools a new tool is run only long enough to identify the first stages of deterioration. The existing database and instrumented machines now can provide a major resource for evaluating new tool treatments such as implantation. The capability exists to do this much more rapidly and without operator bias vis-a-vis standard statistical methods.

The meeting agenda then turned from individual tools/techniques applications to descriptions of technology transfer, mechanisms, resources for technology transfer and small business ion implantation options. Mr. George Terrell of Headquarters, US Army Materiel Command, described the environmental problems facing the Army and MANTECH (Micro-factory) approach to solving these problems. This program would, ostensibly with the advice/oversight of an expert advisory group, fully validate environmentally acceptable production line output of a needed product relative to surface treatments (coating, stripping, cleaning etc.). Currently the Army was involved with approximately $28.4 Billion/yr. expenditures for hardware. For many situations introduction of new technology with the microfactory concept could have direct impacts on such future investments. An example of a high visibility area where environmental concerns mandate change is current use of cadmium plating. Similarly Mr. W.E. Barkman reviewed DOE cooperation within their Precision Flexible Manufacturing Systems (PFMS) concept. This involves nuclear weapons production plants, NIST and US private industry.

The objective of this program is quality manufacture through process certification rather than product certification.

Dr. Ralph Alexander, President of Ion Surface Technology Inc., followed with a summary of market barriers for a small company. The major barrier is still cost. He noted that industry is conservative and frequently will not utilize treatments where the treatment costs more than 30% of the part. This despite the fact that life cycle cost savings might be on the order of 200 or more percent. Currently ion implantation is not perceived to be competitive with other surface treatments. Several recent technical developments that could greatly increase market acceptance are PSII processing (as discussed earlier Conrad, Roth), larger scale implanters (reduced cost/part), and ion assisted coating techniques. In the ensuing discussion it was mentioned that some of these larger systems are already available in England. Alexander also pointed out that nitrogen implantation was not a cure-all and for shallow penetration depths it is not retained in steel above 350°C. Also, one must understand the function of the part as the retained dose depends on part geometry. This makes uniform implantation difficult on complex surfaces. Such variability can sometimes affect product performance. PSII has an advantage relative to beamline ion implantation in this regard.

Dr. Howard E. Clark, Director of Research for the American Society of Mechanical Engineers (ASME) ended up the formal talks with an overview of support mechanisms for Emerging Technology standards development. He gave a listing of governmental and industrial groups that the ASME had worked with or was currently assisting. Typical activities organized could involve getting together a topnotch technical advisory group to review capabilities and types of equipment needed to carry out a national effort. He thought it would be completely feasible to be contracted for an ASME reviewed assessment of National ion implantation equipment needs.

After the conclusion of the formal talks a panel was convened with Dr. James McCauley, Moderator and Dr. Raymond Buchanan, Secretary. The Panel Members represented a cross-section of users and suppliers of ion beam implantation technology.

PROGRAM

WORKSHOP ON RESEARCH AND TECHNICAL TRANSFER OF ION IMPLANTATION TECHNOLOGY FOR SPECIALTY METALS

Hyatt-Regency Hotel, Knoxville, Tennessee

October 25-27, 1989

Dr. Robert R. Reeber, Workshop Chairman
Dr. J.Reece Roth, Local Arrangements Chairman
Dr. Raymond A. Buchanan, Scientific Secretary

Thursday, October 26, 1989

Welcome and Introduction:

8:30 Welcome: Dr. Thomas C. Collins, Vice-Provost for Research, University of Tennessee, Knoxville

8:40 Meeting Objectives: Dr. Robert R. Reeber, ARO

Research in Progress: Dr. Robert R. Reeber

9:00 Keynote: Eighteen years of Ion Implantation Technology with Speciality Materials, Dr. Geoff Dearnaley, Harwell

9:50 Corrosion Inhibition by Plasma Ion Implantation, Professors J.Reece Roth and Raymond A. Buchanan, University of Tennessee, Knoxville

10:35 Plasma Source Ion Implantation for Wear and Tool Applications, Professor John Conrad, University of Wisconsin, Madison

11:15 High Current Metal Ion Source for Ion Beam Implantation, Dr. Ian Brown, Lawrence Berkeley Laboratory

Government/Industry Experience: Dr. James Mayer, Cornell University

1:00 Ion Implantation Research at Oak Ridge National Laboratory, Dr. J.M. Williams

1:30 Ion Beam Implantation Research-Specialty Materials Applications, Dr. Graham Hubler, Naval Research Laboratory

2:00 Ion Implantation Production and Quality Control Experience, Ray Bricault, Spire Corp.

Thursday, October 26, 1989 (cont.)

2:30 Coffee with Supplemental Poster and Exhibit Session

Army Experience: Dr. Graham Hubler, NRL

3:10 Army Experience with Ion Implantation of Cutting Tools in the Production Environment, Dr. Robert Culbertson, MTL

3:35 CCAD Experiences with Ion Implantation, Mr. Alonzo Gonzales, Corpus Christi Army Depot

4:00 DERSO Helicopter Problems, Dr. Lewis Neri, Depot Engineering and RCM Support Office, Corpus Christi Army Depot

4:30 Methods for Rotary Cutting Tool Evaluation, Dr. Jack Moriarty, Rock Island Arsenal

4:55 Adjourn

Friday, October 27, 1989

Technology Transfer Issues: Dr. Robert Culbertson, MTL

8:15 Environmentally Acceptable Metal Treatment Processes, Mr. George Terrell, U.S. Army Material Command

8:45 D.O.E. Production Needs for Improved Performance, Mr. Fred Jones, Director, Advanced Manufacturing Technology, Martin Marietta Energy Systems

9:15 Ion Implantation Technology Options from the Perspective of a Small Business, Dr. Ralph B. Alexander, President, Ion Surface Technology, Inc.

9:45 Support Mechanisms for Standards Development in Emerging Technologies, Dr. Howard Clark, Director of Research, ASME

10:15 Coffee

Industry (Users and Suppliers)/Government and Academic Panel
Moderator-Dr. James McCauley, MTL; Scientific Secretary-Dr. Raymond A. Buchanan, University of Tennessee, Knoxville

Status and Prospects for Commercial Standards and Specifictions for Ion Beam Treatments of Tools/Bearings and Specialty Metals. State of the Technology, Assessment of Barriers to Implementation and Recommendations for Technology Transfer and Implementation.

Friday, October 27, 1990 (cont.)

10:45 Opening Remarks

1. Experience with Mantech Program, Spire Corp.
2. Perspective from a Helicopter Company
3. Tool and Die Industry Representative
4. Ion Beam Company Comments

11:25 General Discussion/Comments/Recommendations

12:30 Adjournment

MEETING OBJECTIVES

Dr. Robert R. Reeber, Workshop Chairman
Army Research Office
Materials Division
4300 S. Miami Blvd.
Research Triangle Park, NC 27709

The objective of this meeting is to provide industry (suppliers and users), researchers, and government a forum for identification of the specifics required to transfer appropriate ion implantation technology for private and government use. It is hoped that recommendations provided here will significantly hasten commercial specifications for this emerging technology.

A secondary objective is to highlight new research that can provide increased capabilities in the future.

18 YEARS OF ION IMPLANTATION TECHNOLOGY WITH SPECIALTY MATERIALS

Dr. Geoff Dearnaley
Harwell Laboratory, Didcot, U.K. 0X11 ORA

ABSTRACT

The application of ion implantation to materials other than semiconductors commenced around 1970 with the goal of achieving improvements in their tribological and corrosion properties. It was soon established that nitrogen would increase wear resistance and yttrium would increase the oxidation resistance of a wide range of steels and other alloys. The mechanisms responsible for these effects are complex, but have become increasingly well understood as a result of the research carried out over subsequent years, and the latest ideas will be summarized.

Some of the most sophisticated implantation treatments exploit the non-equilibrium nature of the process. For instance, it is possible to introduce over-sized atomic species without the usual constraints of solubility or diffusivity. Surfaces can be rendered amorphous by the implantation of appropriate metallic or metalloid species, and the resulting freedom from grain boundaries conveys low friction and good corrosion resistance.

Examples of the successful application of ion implantation to metallic components such as tools, bearings and medical products will be presented, together with an outline of the development of equipment for the application of the process on a production scale and at the most economic cost.

Ion implantation has a number of striking technical advantages, such as the low-temperature nature of the process, its versatility and controllability. However, the depth to which a material can economically be modified is limited to a fraction of a micron, and there is not complete control over the surface composition.

For this reason, a treatment combining physical vapor deposition with ion implantation, known as 'ion beam assisted deposition' or 'ion assisted coating' has been the subject of much recent research. Some products utilizing it are already on the market. Compact, highly adherent coatings of a wide range of composition has been produced in this way, again at low process temperatures. These comprise metallic or ceramic coatings some of which provide excellent wear resistance, while others are remarkably resistant to aqueous corrosion and to oxidation. Recent research will be reported, and suggestions made as to how the process may be further developed to achieve the maximum degree of protection for critical components.

In summary, ion beam technology has been developed over the years to the point at which it is now possible to tailor surface composition and microstructure in order to meet a wide variety of requirements in field applications. The methods described are especially suitable for the treatment of precision components in order to achieve a longer service life.

CORROSION-INHIBITION BY PLASMA ION IMPLANTATION*

J. Reece Roth and Philip F. Keebler
UTK Plasma Science Laboratory
Department of Electrical and Computer Engineering

and

Raymond A. Buchanan and In-Seop Lee
Center for Materials Processing
Department of Materials Science and Engineering
University of Tennessee
Knoxville, TN 37996-2100

ABSTRACT

It has been well established that the implantation of nitrogen ions having energies above 20 keV can have a beneficial effect on the hardness and wear resistance of metals of engineering interest. This paper is a preliminary report on the corrosion related characteristics of 304L stainless steel samples which have been implanted with nitrogen ions having energies of 20 keV, using plasma ion implantation techniques in a steady-state Penning discharge. Plasma ion implantation offers many potential advantages over the widely-used ion beam implantation, including short exposure times (on the order of minutes), isotropic incidence of the ions, and the ability to uniformly implant complex surfaces such as screw threads, gear teeth, and turbine blades.

A principal objective of this research effort was to develop a fast, high voltage switch capable of applying up to 50 kV to the sample for periods of ten microseconds or so, and then rapidly turning off the voltage for a duty cycle of several times the period of voltage application. These short duty cycles allow the required fluence of ions (ions/cm^2) to build up in a few minutes, compared with exposure times on the order of an hour associated with long duty cycle switches based on spark gap technology. Another objective related to the technology of plasma ion implantation was development of electrical instrumentation for the current and voltage applied to the sample such that the total ion fluence and characteristic ion energies could be measured during the entire sample exposure time.

The metallurgical objectives of this work include the evaluation of nitrogen concentration profiles in plasma-implanted Type 304L stainless steel, and subsequent electrochemical evaluation of any resulting inhibition of chloride-induced pitting corrosion. We expect to present results from implanted samples and unimplanted control samples which have been electrochemically characterized in terms of anodic and cathodic polarization behavior, uniform corrosion intensity, critical pitting or crevice corrosion potential, and protection potential.

*Supported in part by ARO contract DAAL03-88-K-0161, in part by the UTK Center for Materials Processing and the Department of Materials Science and Engineering, and in part by ARO Grant DAAL03-89-G-0125.

PLASMA SOURCE ION IMPLANTATION FOR WEAR AND TOOL APPLICATIONS

J. R. Conrad, University of Wisconsin, Madison

ABSTRACT

Plasma Source Ion Implantation (PSII) is a non-line-of-sight technique for surface modification of materials. Targets to be implanted are placed in a plasma chamber and are then pulse-biased to high negative voltage (10-100 kV). A plasma sheath forms around the target, and ions bombard the target from all sides simultaneously without target manipulation. PSII minimizes the problems of shadowing and excessive sputtering of the target material, which can severely limit the retained dose. PSII has demonstrated (1) efficient implantation of ions to concentrations and depths required for surface modification, (2) dramatic improvement in the life of manufacturing tools and manufactured components in industrial applications, (3) acceptable dose uniformity on non-planar targets without target manipulation, and (4) that such uniformity can be achieved in a batch processing mode.

Most of our research to date has been limited to nitrogen ion implantation, which, for example, is often effective in increasing the surface hardness and wear resistance of steels with high chromium content in wear applications at low to moderate temperature. While some of the materials problems of industrial interest are amenable to nitrogen implantation with our present PSII facility, the facility is unable to address applications such as: (1) surface hardness and wear resistance of low chromium steels, (2) surface hardness and wear resistance of ceramics and other non-steels, (3) hardness and wear in high temperature environments, (4) corrosion resistance, and (5) oxidation resistance. For these broader applications, ion species such as titanium, carbon, chromium, etc. will be required. To address these problems, we are extending the PSII process to operate in a molecular ion species mode, and modes similar to the IBED (Ion Beam Enhanced Deposition) or ion beam mixing techniques which have been developed using conventional technology.

In this presentation we will discuss (1) the nitrogen ion PSII laboratory and field test results, (2) Monte Carlo simulations and experimental results on species treated by PSII in ion beam enhanced deposition mode in our current PSII system, and (3) the construction status of a substantially larger, more versatile PSII system which will allow IBED operation in addition to nitrogen ion implantation.

HIGH CURRENT METAL ION SOURCE FOR ION IMPLANTATION

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Berkeley, CA 94720

A high current metal ion source has been developed in which a metal vapor vacuum arc is used as the plasma discharge mechanism, by means of which high current beams of a wide range of multiply-charged metal ions can be produced. Present embodiments of the source operate in a pulsed mode, with pulse width of order 1 ms and repetition rate up to 100 pps. Beam extraction voltage is up to 100 kV and the peak beam current is up to several Amperes. The ions produced by the vacuum arc discharge are typically multiply charged with a mean charge state of 2 to 3, and thus the mean beam energy can be up to several hundred keV. The source has been operated with virtually all of the solid metals of the Periodic Table.

Several different versions of the metal vapor vacuum arc ion source concept have been developed, with features such as a multiple cathode configuration whereby one can switch rapidly between up to 18 different metal ion species, and a broad-beam (10 cm diameter) extractor.

The source has been used for a number of different high dose metal ion implantation research applications, as well as for injection of high current metal ion beams into heavy ion synchrotrons for basic nuclear physics research, at several different laboratories around the world. Implantation applications that are being pursued at LBL include metallurgical surface modification for improved tribological properties (friction, wear, hardness) and high temperature oxidation resistance, and 'fine tuning' of the composition of high-Tc superconducting thin films.

In this paper the metal vapor vacuum arc ion source concept and performance characteristics are described, and the metallurgical ion implantation research programs being carried out at LBL are summarized. The importance of this technology to very large scale metallurgical surface modification is reviewed.

ION IMPLANTATION RESEARCH AT OAK RIDGE NATIONAL LABORATORY*

J. M. Williams, C. M. Egert,[a] C. J. McHargue, R. A. Buchanan,[b]
D. K. Thomas, W. C. Oliver, and R. H. Staunton
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

This presentation will survey ion implantation programs at Oak Ridge National Laboratory that are of interest to the U.S. Army. In the area of specialty metals, research includes studies on ion implantation of U, Be, Ti, Ti-6Al-4V alloy, and hard chrome plate. An extensive survey on ion implantation of various elements into U for protection against corrosion in water vapor was done. In addition to the chemical effects produced by the implanted constituent, the ion implantation parameters of range and sputtering will be of major importance in process design for use of ion implantation in corrosion protection of U. It would appear that ion implantation treatments which are superior to the best ion-plated Al coatings can be designed. The results are of possible interest for penetrators. Strategies and results for smoothing and cleaning of Be mirrors by use of ion beams will be described. Research on ion implantation of surgical Ti alloy has resulted in large improvements in wear under conditions applicable for orthopedic prosthetic devices; the process is now fully commercial for artificial hip joints, knee joints, and other appliances. Recent efforts have concentrated on implantation of Ir ions into Ti substrates for biocorrosion inhibition and for production of iridium oxide films. Iridium oxide itself can be considered to be an electronic device, in that the material has favorable "charge injection" or electrolytic capacitance properties. Research in ceramic materials has been directed in part toward hardening and improvement of engineering fracture stress in Al_2O_3. Favorable results have been obtained, particularly for direct ion implantation of Cr into sapphire. These results are of interest for IR windows and production tooling. Ion implantation and correlated annealing studies have been performed on $LiNbO_3$ substrates for production of waveguide devices. A program of failure analysis of helicopter components has existed; many failures are due to problems with corrosion, tribology, and pertinent coatings and surface treatment techniques. Possible applications for ion implantation in these areas will be identified.

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ION BEAM IMPLANTATION RESEARCH-SPECIALTY MATERIALS APPLICATIONS

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ABSTRACT

Several examples of applications of ion implantation and ion beam assisted deposition (IBAD) developed at the Naval Research Laboratory will be given. While ion implantation has had some notable success, a case will be given for the more widespread applicability of IBAD. Examples will include; ion implantation of rolling element bearings, Be gas bearings, Ti and Al metals, and IBAD OF SI_3N_4, BN and TiN. These examples encompass applications to wear and corrosion protection of metals, wear protection of ceramics, and fabrication of optical devices.

ION IMPLANTATION PRODUCTION AND QUALITY CONTROL EXPERIENCE

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ABSTRACT

In recent years, the technology of ion implantation has made the transition from an academic "curiosity", through pilot scale production to that of a full scale production process. A brief review of the changes in the basic form of the ion implanter will be presented, oriented toward paralleling the growth of the technology. A series of production applications will be discussed and the particular aspects of those applications which made them appropriate for ion implantation will be reviewed. The methods presently used in the quality control programs will be reviewed along with a brief overview of yield statistics for components from a number of industries.

ARMY EXPERIENCE WITH ION IMPLANTATION OF CUTTING TOOLS IN THE PRODUCTION ENVIRONMENT

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ABSTRACT

The performance of ion implanted tools vs. unimplanted tools was evaluated in a production machine shop at Corpus Christi Army Depot (CCAD). Taps, reamers, uncoated inserts, and TiN-coated inserts were implanted with nitrogen and in some cases xenon. The implanted tools were not visibly distinguishable from the unimplanted tools. Each tool in this blind study was numerically coded, and the information regarding the ion implantation treatment, if any, was not known by the machine operators. In most tests, state-of-the-art computer numerically controlled machines were used. Standard machine speeds and feed rates were selected for each operation, and the tests were conducted under simulated production conditions. The performance of each tool was based on how many work pieces it was able to machine to within specified tolerances. The results for ion implanted taps and coated inserts showed 1.5 to more than 4 times the performance of unimplanted tools. Tests of reamers and uncoated inserts are in progress, and the evaluation of ion-implanted punch/die sets is underway. The purpose of this work is to demonstrate that ion implantation represents a processing technique that can reduce production costs at CCAD. Ultimately, the goal of this work is to introduce ion implantation processing for helicopter components to enhance the wear and corrosion resistance of flight safety parts and reduce the amount of hazardous waste usage and disposal at CCAD.

CCAD EXPERIENCES WITH ION IMPLANTATION

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ABSTRACT

Ion Implantation is currently being investigated for possible use in the areas of wear, corrosion, erosion, and fatigue of aircraft components and machine tools at CCAD. Since 1986 the Hot Chips and Blue Chips Quality circles have been the driving force at CCAD by justifying small amounts of funding to implant and perform scientific performance testing of machine tools and dies, in actual production runs, with the guidance of the US Army Materials Technology Laboratory (MTL).

The tests were designed to utilize typical machining operations, while maintaining maximum degree of control. The results of the machine tools test will be presented, in which taps and metal cutting Inserts were used on 321 stainless steel, 4140, and 4340 steel hardened to 29 Rc and a repair using ion implanted taps to retap a wielded hole of a aircraft component.

In addition to the tooling test, other ongoing testing using ion implantation such as the results of a punch die used in actual production run to manufacture a locking pin, which used with the assembly of aircraft turbine blades. The die has shown considerable amount of extend wear life when compared to past job runs, as well as other interesting observations. The need to look in the future for tooling capable of cutting hardened and exotic materials with less down time due to variables encountered in production that are not seen in basic research.

Ion Implantation is also being investigated in the area of chrome plated aircraft parts in an attempt to reduce the need of reworking certain aircraft components requiring chrome plating, as well as several parts that flow through the cadmium line in which hazardous chemicals used in the solutions and disposal costs are expensive.

DERSO HELICOPTER PROBLEMS

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ABSTRACT

This presentation is a brief overview of the Depot Engineering and Reliability Centered Maintenance (RCM) Support Office's (DERSO's) capabilities and current operations as they relate to corrosion and wear, and the potential application of this new technology to some of DERSO's helicopter problems whose time has come. In this presentation, Dr. Neri will emphasize the main function of his office in the support of the Corpus Christi Army Depot (CCAD) at Corpus Christi, Texas, and the U.S. Army Aviation Systems Command (AVSCOM), St. Louis, Missouri. As a significant element of their work, DERSO makes engineering decisions on the usability of "aging" aircraft components.

DERSO helicopter problems are many. However, they all fall either in the power plant or airframe area.

The presentation will primarily focus on the significant few and not the insignificant many of the Army's helicopter problems at the depot. It should be noted that DERSO has the technical responsibility for the technical content of the depot maintenance work requirements (DMWRs), both in-house and outside the depot.

A DMWR is a comprehensive document which contains complete overhaul criteria, identifies minimum acceptable standards, and, where applicable, provides preshop analysis guidelines for determining the extent of repair required. it is normally provided as the "statement of work" for each item contracted or programmed for depot level maintenance. It is a "how to do" type of document which provides the necessary instructions for the complete overhaul of the item, including modification of parts, and assemblies and/or parts, subassemblies, and assemblies required to convert to latest item configuration as specified in depot program notices.

DMWRs are supplemented in the depot by AVSCOM Engineering Directives (AEDs). AEDs address specified problems in a DMWR and serve as an aid in updating the DMWR. AEDs are also used to formulate technical data packages for piece part repair contracts and provide alternate procedures to the depot because of unique capabilities or restrictions. The DMWR will be the vehicle by which this new ion implantation technology would be implemented by DERSO at CCAD.

METHOD FOR ROTARY CUTTING TOOL EVALUATION

By

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ABSTRACT

Implementation of unattended machining systems and flexible manufacturing cells demand high reliability cutting tools with predictable wear life. Technology was developed and is in use at Rock Island Arsenal (RIA) to capture and record certain dynamic power data during cutting sequences. This record dynamic data feature, also known as "Freeze-Frame" capability, provides printouts of machine horsepower, spindle speed, feed rate, and axial thrusts. This information allows the calculation of specific energy (unit horsepower/in^3/minute) and other means for determining tool wear rates and discerning trends. It has been possible to evaluate the behavior of coated carbide inserts, various supplier HSS drills, different repoint geometries, unique thermal treatments, and special design/configuration rotary cutting tools. Extensive tool tests employing end mills and drills were conducted in RIA's main production machine shop during routine machining of prismatic parts. Special command program tapes were prepared for use on six upgraded CNC horizontal machining centers. Test data collection causes no interruptions during machining cycles nor are any undue process delays experienced. Cost and productivity algorithms were utilized to compare the performance of new and reground tools.

ENVIRONMENTALLY ACCEPTABLE MATERIAL TREATMENT PROCESSES

George H. Terrell
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ABSTRACT

DEFINITION. The Environmentally Acceptable Material Treatment Processes (EAMTP) MANTECH Thrust will establish and maintain for the Army Materiel Command (AMC), the technical organization and capabilities to ensure environmental and worker safety regulatory compliance. This organization and capabilities will provide the means for AMC to validate or develop, proveout and implement, new and/or modified materials, treatments, manufacturing processes and process controls which reduce air, water and solid pollution, ensure environmental regulatory compliance, and increase worker safety while maintaining current industrial capability. The scope of the effort will include all surface finishes, treatments and processes used on composites, electronics, and metals for all Army material.

ORGANIZATIONS.

A. (Core Team) AMCPD-BD, MTL, BRDEC, PDMA, ARDEC, ARINC Research, Inc., Ocean City Research.
B. (Army) ASA (I&L), OTSG, AEHA, USATHAMA, AMC Surgeon, AMCEN (HAZMIN).
C. (Navy) NAVAIR, NAVSEA
D. (Air Force) AFSC, AFLC.
E. (DOD) DLA.
F. (Federal) DOT, DOE, DOC (Coast Guard), EPA, OSHA, NIST.
G. (Industry) Various professional and technical associations.

MICRO-FACTORY POTENTIAL

A. The micro-factory is a business organization established under the auspices of this Thrust program. The operating principle is to provide resources (in a cost sharing venue) to DA, DOD, other Federal agencies, and industry for the validation/development, improvement, and production testing of material treatment processes that minimize or eliminate unacceptable environmental discharges (air, water, ground). The desired result of any micro-factory project will be environmentally compliant processes, process controls, and products meeting or exceeding the operational capabilities of current treatment processes.

B. The principal products of the micro-factory will be fully validated/developed, production line tested, documented environmentally acceptable material treatment processes relative to surface treatment (coating, stripping, cleaning). The micro-factory will provide equipment to model production use of a process/process controls; personnel to run the process, conduct the process evaluation; and engineering support to prepare equipment requirements, plan shop floor designs, technical instructions,

training curricula, transition plan (to full scale manufacturing floor), and other documentation necessary for use in manufacturing and maintaining government material.

PRIORITIZED SUBTASKS. The Core Team, with the advice of an Advisory Group, will select and prioritize projects for demonstration within the micro-factory concept. Projects will include the removal of, or identifying alternatives for volatile organic compounds (VOC) contained in paints, coatings, and other solvent containing materials; chlorofluoro-hydrocarbons (CFC), chlorinated hydrocarbons, and HALONS; and recovering or eliminating the polluted water and solid wastes associated with plating, stripping and cleaning operations. Micro-factory operations will peak at $7M and are expected to stabilize at $6M (per year).

DOE PRODUCTION NEEDS FOR IMPROVED PERFORMANCE

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ABSTRACT

In October 1986, the U.S. Department of Energy/Albuquerque Operations Office (DOE/AL) established a cooperative Precision Flexible Manufacturing Systems (PFMS) effort among the nuclear weapons production plants; design laboratories; the MBS, now National Institute of Standards and Technology (NIST); and U.S. private industry to develop and implement critical technologies for the remote manufacturing and in-process certification of precision hemishell components. The DOE structured the program in three phases: (1) critical technology demonstration; (2) prototype demonstration; and (3) remote manufacturing implementation.

The long-range PFMS goal is to provide technology for the remote fabrication of precision hemishell components from toxic, radioactive and hazardous materials. Embodied in this long-range goal are two objectives that are driving the program; these are the contour tolerance and the process certification requirements. The quality objective is to manufacture quality into the product rather than inspect quality into the product by certifying the process rather than the product.

ION IMPLANTATION TECHNOLOGY OPTIONS

FROM THE PERSPECTIVE OF A SMALL BUSINESS

Ralph B. Alexander

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Ion implantation has been used for surface modification of metals in industrial applications since the process was developed at the UK Harwell Laboratory in the early 1970's. However, the market for ion implantation of metals, especially nitrogen implantation to improve wear and fatigue resistance, has been limited so far. Both market and technical barriers to more widespread acceptance of the technology in industry will be discussed.

Of market factors, the biggest barrier at present is cost. A frequent requirement in industry is that the cost of ion implantation be no more than about 30% of the cost of the item implanted. A second cost requirement is that ion implantation be competitive with other surface treatments. Both these requirements are difficult to meet for small tools and components. Other market barriers include industrial conservatism and production priorities in manufacturing.

Technical factors include the size of available implanters, the line-of-sight restriction of conventional beamline implantation, sputtering, and other process limitations such as shallow penetration depth. The size limitation restricts the capabilities for implanting both large items and large batches of small items. This and the line-of-sight limitation have an important impact on the cost of ion implantation.

Several recent technical developments that should greatly increase market acceptance will be discussed from the perspective of a small business. These are: (1) the non-line-of-sight plasma source ion implantation (PSII) process, (2) large-scale nitrogen implanters, and (3) ion assisted coating techniques. Large scale implanters will reduce the unit cost of ion implantation, while ion assisted coatings will considerably widen the market. For nitrogen implantation, the most promising development is PSII which overcomes the line-of-sight and sputtering limitations of beamline implantation, and can be readily scaled up. Both the capital cost of a production PSII implanter and the processing cost will be lower than for a comparable beamline machine.

SUPPORT MECHANISMS FOR STANDARDS DEVELOPMENT IN EMERGING TECHNOLOGIES

Howard E. Clark
Director of Research
American Society of Mechanical Engineers

ABSTRACT

For well over 100 years, this nation's professional societies and trade associations have been involved in the development of codes and standards -- procedures that specify how materials and products are to be manufactured or tested as well as the ways in which systems and structures are to be built. Throughout this period, the American Society of Mechanical Engineers has been a leader in developing new standards or revising existing ones in response to emerging technologies and changing needs.

One reason for the Society's success in meeting these challenges has been the availability, for over 80 years, of a formal, Society-organized research program. The function is now carried out by the ASME Center for Research and Technology Development in Washington, DC. The Center, which was established in 1985, provides a cost-effective mechanism for generating new information needed for the development of new standards or for the revision of existing ones. Additionally, the Center offers a mechanism for drawing together existing data from diverse sources and converting them to forms that are more useful both to standards writers and to practicing engineers.

Traditionally, this service has been used primarily to support internal, ASME standards development efforts. However, since the Center was established in 1985, ASME has made a conscious effort to make the Center's services available to external groups.

Dr. Clark will review the kinds of support services that the Center offers and explain how they could be made available to committees seeking to develop procurement standards for materials treated by ion implantation technologies.

Supplemental Program

A TIME-OF-FLIGHT TECHNIQUE FOR THIN FILM ANALYSIS BY MEDIUM ENERGY ION SCATTERING

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ABSTRACT

We describe a high resolution time-of-flight technique for surface analysis by medium energy ion scattering (MEIS). Using ions in the 100-500 keV energy range, this technique is capable of providing sensitivity and depth resolution equaling or exceeding that of conventional MeV Rutherford backscattering spectrometry (RBS) performed with a surface barrier detector. The resolution of the time-of-flight detector is between 140 and 200 ps and the effective solid angle is about 3×10^{-5} sr. We report measurements of the detector efficiency as a function of energy for α particles and provide examples of the detector performance analyzing thin films by α backscattering, Li^+ backscattering, C^{++} backscattering, and (forward) elastic recoil detection. The latter technique promises to be an excellent way to detect surface hydrogen and may be able to provide hydrogen depth profiles in the near surface region.

MINUTES

of

INDUSTRY (USERS AND SUPPLIERS), GOVERNMENT, NATIONAL LABORATORY AND ACADEMIC PANEL DISCUSSION

Panel Members:

Mr. George Terrell, U.S. Army Materiel Command
Dr. Lewis Neri, Chief, Depot Engineering and RCM Support Office/AVSCOM, Corpus Christi Army Depot
Mr. Ray Bricault, Spire Corporation
Dr. A.J. Armini, Implant Sciences Corporation
Dr. Edward B. Hale, University of Missouri-Rolla
Dr. Howard E. Clark, Director of Research, American Society of Mechanical Engineers

Dr. McCauley initiated the forum with a short list of possible discussion issues: (1) the perceived barriers to implementation of ion implantation technologies, including the technology push/pull issue, psychological issues, competing technologies and relative costs; (2) implementation options available, and specifically who would accomplish the ion implantation of tools and aircraft components, ie. job shops, tool/component manufacturers, the Army with on-site ion implantation machines, or a combination of these possibilities; and (3) an assessment of the meeting including subsequent actions and or recommendations that might be appropriate results of the workshop. He briefly pointed out the rapid progress that Japan was making in this area with a major industry/government coordination center (67 member organizations) in Osaka. This organization, acronym AMMTRA, is conservatively spending $110 million per year for ion implantation, new equipment development, and related materials science, etc.

The panel members each presented views before the forum was opened up for general discussion.

Mr. Terrell addressed an issue of importance in the Army MANTECH program relative to the dichotomy between free transfer of information from ion implantation companies and protection of their proprietary rights. Under the program, proprietary company information would not be placed in the public domain until that information had been verified through production-type testing procedures over a 2-3 year time period. In answer to a question from the audience regarding the mini-factory concept and how it would be implemented for ion implantation technology, he indicated that it most probably would be a combination of approaches depending on the particular application. Some work might be

farmed out to service companies while other work could conceivably be done through cooperative efforts with Army personnel.

Dr. Neri reiterated his goal of achieving the necessary Army support for implementing ion implantation processing at the Corpus Christi Army Depot, and using this site as a "showcase" for other Army facilities.

Dr. Armini briefly presented ion implantation capabilities at Implant Sciences Corporation, and then discussed the difficulties encountered in selling ion implantation to large American industries. His company, Implant Sciences Corporation, could be considered typical of the U.S. ion implantation industry. About 90% of their work is service oriented. They operate 6 implanters, four of which have capability for mass analysis. Over the years they have been in operation they have developed extensive capabilities and fixtures for part manipulation. About 10% of their business is with the government. Their work has involved solid lubricants and ceramic implants. They are involved with production processes for space shuttle parts and prosthetics. These parts range in cost from $100 to $500/part. In development they are addressing providing services for $50/part aircraft bearings, gears and tooling. Basic research is aimed at providing cost-effective treatments for $5 parts.

Mr. Bricault reviewed the Spire Corporation experience with the Navy MANTECH program. He found the program of value for establishing a production ion implantation facility and production procedures. (A comment was made from the audience that one of the best results from the Navy MANTECH program was demonstration that the technology was industrially feasible.)

Dr. Clark summarized the possible role the ASME Center for Research and Technology Development could play in the technology transfer issue. Some possibilities included: (1) organization of a committee of experts to assess the state-of-knowledge of ion implantation technology relevant to industrial production situations--the report could be quite useful in increasing the industrial awareness of the technology, and (2) organization of efforts to have ASME codes and standards developed for ion implantation processing.

Dr. Hale reviewed the program for the up-coming "Workshop on Applied Ion Implantation," St. Louis, Missouri, November 16-17, 1989. The workshop will emphasize industrial applications, rather than research results, and the program includes numerous reports by industrial users worldwide.

The program was opened for general audience participation, and a number of issues were raised.

A discussion was initiated on the advantages of ion-beam implantation (line-of-sight, amenable to magnetic beam purification) versus the emerging PSII method (amenable to complex shapes without workpiece manipulation). It was generally agreed that the most advantageous method would depend on the specific application.

The issue of technology push/pull, i.e. push from the developers/researchers/ion-implantation-companies versus pull from potential industrial users, was discussed. The consensus was that considerably more push than pull existed at the present time. Given that ion implantation is a proven technology now for many applications, reasons were offered for the lethargic industrial response. Possible reasons were: (1) perceptions that the benefits are insufficiently large to merit the change-over efforts, (2) unawareness of production-machine availability, (3) perceptions that prohibitively high costs are involved and that the improved items would simply not sell, and (4) concern that information on processing parameters is proprietary and not available.

The question was raised: "Where are the large American industries in the overall development and utilization of ion-implantation technologies?" It was observed that in other countries (Japan, France, England), processing for internal use or external marketing is associated with major industrial corporations. Whereas, in this country, ion implantation is a small business activity which often must go to foreign investors for financing.

It was observed that in Japan a large government investment into ion implantation technologies ($800M to $1B over a few years) is being made with industrial/academic cooperation. Statements were made that such a national focus was needed in this country, with emphasis on goals, strategies, state-of-the-art assessment of industrial problems that can be solved with ion implantation technologies today, a status report on industrial equipment currently available, and identification of needed industrial development. With the current emphasis in foreign countries, and lack of large industry involvement in this country, it was speculated that within 10 years probably all production type implantation equipment will be manufactured abroad; and again, this country will lose the advantage in a major advancing technology. A reference was made to the difficulty in obtaining money for production-type ion implantation equipment development. To some extent the problems were similar to early American developments of electron microscopes and currently with lithography equipment at Perkin Elmer. Overall the conferees said they felt the workshop was very timely and had focussed on some important issues. The timing was felt to be right to push for more widespread use of ion implantation technology.

Statements were made that for significant American-industry commitment to ion-implantation technologies, pilot-plant results (not more laboratory results) were needed. Such industrial data as that now being collected at the Corpus Christi Army Depot would be most convincing.

It was observed that environmental concerns and regulations are placing increasing restrictions on the use of electrodeposition technologies, and that these events could force industry to adopt "clean" surface-modification techniques such as ion implantation. These environmental considerations may provide an initial driving force toward large scale commercialization of ion implantation.

Research and Technical Transfer Review of Ion Implantation Technology for Specialty Metals

October 26-27, 1989 ** Hyatt Regency Hotel ** Knoxville, Tennessee

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